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EVALUATION OF A MANUFACTURING PROCESS FOR PRODUCING PYROLYTIC GRAPHITE INSULATING SLEEVE OF MAXIMUM STRUCTURAL INTEGRITY

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(NASA-CR-75831) EVALUATION OF A MANUFACTURING PROCESS FOR PRODUCING PYROLYTIC GRAPHITE INSULATING SLEEVE OF MAXIMUM STRUCTURAL INTEGRITY (Westinghouse Astronuclear Lab., Pittsburgh) ~~24~~ 25 p

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EVALUATION OF A MANUFACTURING PROCESS FOR PRODUCING PYROLYTIC GRAPHITE INSULATING SLEEVE OF MAXIMUM STRUCTURAL INTEGRITY

[U]

INFORMATION CATEGORY

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Manufacturing Engineering

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ABSTRACT

An evaluation was made of a new concept manufacturing process for producing an insulating sleeve of maximum structural integrity. One furnace run consisting of 50 Tapered Wall As-Deposited Pyrolytic Graphite Bats deposited to a 9" length, yielded without any O.D. or I.D. machining, 46 functionally usable insulating sleeves. No delaminations intersected either unmachined surface, thermal conductivity appeared to be adequately low, and brushing and component vibratory testing established that this high yield processed sleeve is of maximum physical integrity. Manufacturing and Supplier Cost estimates indicate that this manufacturing process should result in an substantial cost reduction.

INTRODUCTION

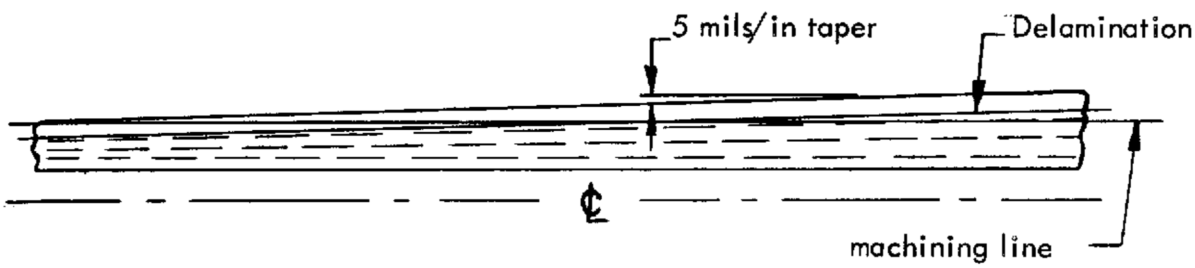
A major problem in the manufacture of Insulating Sleeves is the intersections of delaminations with the finish machined O.D. and I.D. surfaces. These intersections are a parting of the material seriously reducing the physical integrity of the sleeve. They result in spalled areas of varying depth and may encompass a third or more of the sleeve's surface. (See Fig. 3) The machined surface invariably intersects at least one delamination which results in components subject to further spalling and flaking during inspection, reactor assembly, and operation. Substantial improvements have been realized in the physical integrity of the conventionally manufacture sleeve by controlling the type of microstructure, but the optimum of quality has not yet been achieved because of the inherent combination of the material's residual stresses and the "ball bat effect" resulting from the standard deposition process.

The residual stresses which arise in the fabrication of a cylindrical pyrolytic graphite shape are due to the interacting phenoma of the material's anisotropy and growth stresses. The anistropic stresses are due to the large difference in the "a" and "c" directional expansion coefficients and are applied during the cool down from deposition temperature to room temperature. When the thickness to radius ratio (t/r) of cylinders is small (less than 0.10), the stresses can be considered to be biaxial since the radial component is negligible. When they exceed the strength of the material, the material failure is usually by I.D. longitudinal cracking and occasionally by I.D. helical cracks (caused by biaxial stresses resolved into shear). In the case of large t/r's such as the subject sleeves, the radial stress becomes controlling since the "c" direction tensile strength is the weakest and failure of the solid wall is by excessive delamination into thin concentric shells.

The growth stresses are due to the phenomena of lattice transformation or graphitization of layers as they are deposited. The graphitization and annealing amounts to an ordering effect on the atomic scale and is manifested by an irreversible increase (growth) in dimension in the "a" direction and by an irreversible decrease in dimension in the "c" direction.

The magnitude of these stresses because of the large wall thickness to radius ratio (t/r) of sleeves result in a delaminated structure. This is most frequently, a multiple delaminated structure. These delaminations are effective thermal dams, and probably account for the sleeves excellent insulating characteristics.

The manufacturing process for the deposition of the pyrolytic graphite on a male sleeve mandrel results in about a 5 mil/in. taper or "ball bat" deposit. This means that the basal planes have a progressive tapering from O on the I.D. to approximately 5 mils/in. at the O.D. as shown on the following sketch.



Thus it is obvious that the machining line cuts basal planes along the entire length of the sleeve. This cutting of basal planes reduces the components maximum insulating characteristics by heat flow short circuit along the basal planes, but since thermal conductivity testing has established that the sleeve, because of its configuration has insulating characteristics far in excess of the specification, this heat flow consideration is not important. It does result in a sleeve whose surface is conducive to flaking.

One can consider the structure of pyrolytic graphite to be similar to plywood, each layer representing a plane of carbon atoms. Within a layer, the material has excellent strength properties, measuring 10,000 to 20,000 psi, while in the perpendicular direction, the layers are held together by very weak atomic bonding forces, strengths of 500 to 1000 psi being normal. For this reason, delaminations always occur longitudinal to the deposit length, parallel to the orientation of the layers, or basal planes. This is an important consideration only because the machined surface intersects them because of the baseball bat effect. The bat effect is primarily a result of fluid dynamics and the heat transfer characteristics of the gas with respect to the mandrel. Gas depletion, that is dilution of the feed gas by products of reaction, and the variation of gas flow paths are the major causes of the tapered deposits. This tapered deposited tube illustrated in Fig 2, with tapered delaminations is the cause of invariably intersecting delaminations during the machining operation. Result, the present spalled, conducive to additional flaking, insulating sleeves. An O.D. and I.D. brushing technique was recently initiated for determining the acceptability of finish machined sleeves for hot tests. This procedure has resulted in an exceedingly high rate of destructed sleeve. See Fig. 3, 4, 5 and 6.

Corrective action for elimination of the wall taper would require an extensive research and development program for evaluating gas turbulence, gas flows, furnace baffling, and the effects of additives to the gas stream for simultaneously inhibiting and enhancing the deposition. This program would be basic research and development of the pyrolytic process, and accordingly expensive. Another approach would be development of a continuous deposition process, likewise most expensive.

An alternative program is feasible because of the "delamination effect improvement" of the thermal insulating characteristics of pyrolytic graphite in the present sleeve configuration. Southern Research Institute, the sole test facility for thermal conductivity testing for the NERVA Program, has reported sufficient results to establish that P.G. in the sleeve configuration has insulating characteristics at least double that for which the part was designed. Forty-four (44) randomly selected results of tests show an average BTU Ft./Hr. - Ft.^{2} - °F of 0.198 at 3000°F and 0.172 at 2000°F. The specification value (PDS 30014A) requires that the values shall not be in excess of 0.45 and 0.60 respectively. This exceedingly low thermal conductivity permits consideration of utilizing a thin walled or tapered wall insulating sleeve as far as the insulating characteristics only are concerned.

This concept and manufacturing method proposes that tapered wall tubes be deposited to a calculated length such that each deposit will produce without O.D. or I.D. machining, a maximum .005"/" tapered wall sleeve, 4" long cut length, the maximum O.D. at the plane of cutting to be to the present drawing, and a minimum O.D. no more than .020" under this. The resultant as deposited, finished sleeve would have insulating characteristics within the present design requirements of the specification and drawing, and should be functionally satisfactory as no O.D. dimension would be larger and no I.D. dimension would be smaller than that of the present sleeve drawing. This sleeve would be of the maximum structural integrity of the material, as no basal planes or delaminations would intersect a surface, thus no spalling or peeling would occur under normal circumstances.

CONCLUSIONS

1. The tapered wall, as deposited insulating sleeve concept is commercially feasible. This program resulted in yielding 46 sleeves meeting proposed functional tolerances from a furnace run of 50 as deposited pyrolytic graphite tubes.

2. Thermal conductivity of this, varying in cross-section sleeve met the proportionately reduced insulating requirements of this program.

3. This concept sleeve has a maximum of resistance to surface spalling, peeling, and flaking. Vibratory tests and vigorous brushing resulted in no visible surface destruction or dimensional change.

4. It is estimated that procurement cost reduction of approximately 30 to 40 per cent can be anticipated. In addition, a further potential saving may be anticipated because of the elimination of the present 10-15% in-house destructive brushing test rejection rate.

RECOMMENDATIONS

1. It is recommended that Mechanical Design and Reactor Analysis give consideration to the possible application of the tapered wall as-deposited concept insulating sleeve for the NERVA Engine.

2. It is further recommended that the tapered wall sleeve should be clustered, alternately installing the large diameter end towards the nozzle end and then the dome end.

MANUFACTURING PROGRAM

Purchase Order 30484 was awarded to HTM Inc., Lowell, Massachusetts for producing one furnace load of 50 Insulating Sleeves of maximum physical integrity, optimum basal plane orientation and insulating characteristics to Sketch ME-SK-00158, (Fig. 1) this to be accomplished as follows:

1. Deposition process to be that used for current sleeve ordering for deliverable hardware, this to include:
   a. Same size and type of furnace
   b. Same gas components and furnace temperature. Furnace time cycle to be reduced to that which would yield 4" increments to drawing tolerances.
   c. A continuously nucleated microstructure is desired, but not a requirement.

2. The mandrel diameter to be that calculated to produce the I.D. of the sleeve as deposited, to the drawing. A .240" mandrel size was selected.

3. Deposit a minimum of 50, 9" long pyrolytic graphite tubes which will result in a random 4" length meeting the O.D. requirements of ME-SK-00158.

4. No machining or finishing of any kind shall be permitted on the O.D. or I.D. of the as deposited tube.

5. A 4" long sleeve shall be cut from each 9" tube using the $.349 \begin{smallmatrix} +.004 \\ -.000 \end{smallmatrix}$ dimension as the reference for the major diameter.

RESULTS

1. Visual Appearance

The visual appearance of the fifty sleeves was that normal to HTM's current "as deposited bats" excepting the nodule size, which were smaller because of the reduced deposit thickness. There was no evidence of delaminations intersecting either the O.D. or I.D. surfaces. No nodule in excess of 1/16" in diameter was observed. The tubes coloration varied from a dull matte gray to a glossy black. See Fig. 7.

2. Dimensional Inspection

DIMENSIONAL DATA

TABLE I

	Dimension	Drawing Requirement	Quantity to Drawing	Balance
A.	Large O.D.	.349 +.005 -.000	30	13 to .354 - .357 7 ranging .357 - .359
B.	Small O.D.	.314 Min	50	
C.	I.D.	.248 +.006 -.000	46	3 to .245, 1 to .244
D.	Ovality	.003	47	3 tubes .007 - .008
E.	Bow, I.D.	.005	49	1 tube .008
F.	Nodules Height	.005 Max.	43	4 tubes .005-.007, 3 tubes to .007-.011 (.011 nodule located on taper wall close to large diameter, and exceeded the .354" max.

G. Nodules Width	Wall Thickness	46	4 nodules in excess of tapered wall thickness .045, .060, and .075

3. Thermal Conductivity

ACROSS GRAIN THERMAL CONDUCTIVITY (BTU - Ft./Hr. - Ft.^{2} - $^\circ$F)

TABLE II

Temperature of Test		2000°F	3000°F
PDS Requirement		.60 max.	.45 max.
Project Aim (57% of above)		.34 max.	.25 max.
Test No.	**Tube No.**		
1	4	.13	.17
2	7	.12	.15
3	9	.18	.23
4	19	.09	.12
5	23	.10	.12
6	24	.13	.21
7	25	.16	.17
8	42	.15	.24
Average		.12	.17

4. Macrostructure

A. Longitudinal Cross Section. See Fig. 8 and 9.

B. Diameter Cross Section. See Fig. 10.

5. Microstructure

A. Large Diameter Cross Section. See Fig. 11 and 12.

B. Small Diameter Cross Section. See Fig. 13 and 14

C. As Deposited Nodule, .008" high x .045" wide, see Fig. 15.

6. Physical Integrity

A. All fifty (50) sleeves were cycled five times using the approved WANL Brushing Technique.

No flaking, peeling, or spalling resulted.

B. Thirteen (13) sleeves were clustered by alternately mating the large ends and small ends. The cluster was vibrating tested (E-MIL-44) by Reactor Mechanics.

This simulated core of seven cluster assemblies was laterally vibrated with an MB electro-dynamic shaker from one to five "g's" at various frequencies from 20 to 500 cps. All testing was conducted at ambient conditions.

There was no evidence of flaking or spalling of any of the thirteen sleeves O.D. or I.D. surfaces. The cluster is to be submitted to another cycle of vibration testing. (A very minute amount of powder was present, it being evident that this result from not breaking the sharp corners of edges of the sleeves).

DISCUSSION

1. Visual Appearance

The visual appearance of the sleeves was satisfactory and consistent. Nodule size and frequency approximates that which is normal to the industry. In general nodule size is a function of wall formation thickness, and accordingly any nodules in evidence except at the large end would be progressively smaller. Fig. 15 illustrates an as deposited nodule near the large end whose major axis is slightly larger than the wall thickness. This nodule appears to have been initiated by a soot particle. It illustrates the stressing effect of large nodules and the resultant micro cracking. The industry has the capability of minimizing the frequency of nodules in excess of the wall thickness, but their elimination is not feasible at present. This size and type of nodule is subject to rejection.

2. Microstructure

Figures 11, 12, 13, 14 and 15 illustrate the microstructure at the large and small ends of a sleeve respectively. Since wall thickness is a function of tendency to delaminate there are accordingly fewer delaminations present in the small end of the tapered wall sleeve. The structure is typical of that normally received from this supplier, and in Fig. 13 and 14, illustrates that occasionally a change in furnace conditions occur such as varying gas flows, pressure, or temperature resulting in a change of structure during the deposition cycle.

The inherent characteristics of the tapered wall insulating sleeve with its inherent optimum structural characteristics, downgrade the importance of a highly regenerative microstructure. Marginal and varying regeneration should be acceptable

Since density has primarily been used as an inspection key for determining the to date desired highly regenerative, continuously nucleated microstructure of the machined sleeve, it could be either modified for process control purposes, only, or deleted.

3. Thermal Conductivity (BTU - Ft./Hr. - Ft.^{2} - $^\circ$F)

The across-grain thermal conductivity of the eight sleeves tested averaged .12 and .17 at 2000° and 3000° respectively. For the purposes of this program an aim value of .34 and .25 at 2000° and 3000° was established. These values are 57% of those specified in PDS 30016, which is the ratio of the area at the small of the tapered wall sleeve from that of a machined sleeve. No individual test value was in excess of the aim specified. The T. C. tests were run on the as deposited sleeve with no machining or finishing of any kind permitted. The test specimen were 2" lengths of the sleeve, alternately from the large and small end. The cross sectional area used for computing the thermal conductivity being the average of the areas measured 3/4" from each end.

4. Dimensional

HTM's second furnace attempt, finalizing on a .240" diameter x 9" long mandrel yielded 46 tapered wall sleeves meeting the requirements of the ME-SK-00158 or Drawing 946C055. Results were as follows:

	Large End O. D.	Small End O. D.	I. D.	Bow	Nodules
To ME-SK-00158	30	50	46	49	46
To Dwg. 946C055	20	50	3	--	2
Product yield (Total)	46				

This compares with a 25 to 35 percent yield for the present machined sleeve, irrespective of supplier.

5. Structural Integrity

No surface structural defects were in evidence except for three nodules whose major diameter was in excess of the wall thickness. There was a total absence of surface spalling, peeling, or loose layers. Fig. 7, 8, 9 and 10, compared to Fig. 3, 4, 5 and 6, vividly illustrate the destructiveness of machining small diameter tubes of pyrolytic graphite. Figures 11, 12, 13 and 14 illustrating the pattern of delaminations their being confined within the surface boundaries, and are nearly always concentric to the nearest surface.

No evidence of surface deterioration was evidenced by the cluster lateral vibratory test or by vigorous I.D. and O.D. brushing. Unless there are surface imperfections such as open end basal planes or parting of the planes because of intersecting delaminations, the strength of pyrolytic graphite should be adequate for the sleeve requirement.

The five cycle brushing effected no measurable dimensional change.

6. Economic Consideration

Tapered Wall Sleeve manufacturing and process analysis indicates that a possible 30 - 50 percent cost reduction should result because of the following manufacturing and/or process required changes.

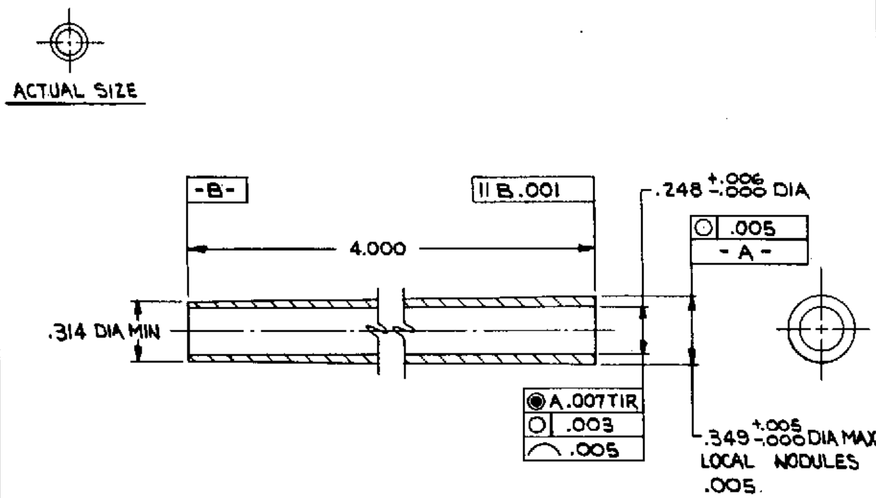
A. Complete elimination of O.D. rough and finish grinding, and I.D. rough and finish reaming.

B. The increase in furnace yield from the present average of 14% of a potential of 120 sleeves, or 17 machined sleeves per furnace run, to a potential of 45 tapered wall sleeves per furnace run, a 260% improvement factor.

C. The improved optimum structural integrity should eliminate the present 10% WANL rejection of sleeves at "brushing" operation.

D. The machined sleeves require an as deposited bat target wall thickness of 0.125 inches in order to obtain two finish machined sleeves from each tapered tube. The tapered wall sleeves target maximum wall thickness is .075 inches. Deposit thickness being directly proportional to deposition cycling time, this permits a 40% reduction in this portion of the furnace cycle. Sleeves are normally deposited at a deposition rate of 5 - 7 mils per hour, the conventional cycle being 20 hours. For the tapered wall sleeve a 7 - 8 hour reduction should result in the furnace cycling of each furnace load.

REVISIONS			
SYM	DESCRIPTION	DATE	APPROVED



QTY REQD	SYM	ITEM NO.	NOMENCLATURE OR DESCRIPTION	CODE IDENT NO.	PART OR IDENTIFYING NO.	MATERIAL	SPECIFICATION	UNIT WT.	ZONE
1		1	SLEEVE		ME-SK00158	PYRO GRAPHITE	PDS30014		

LIST OF MATERIALS

		DRAWN L. MURRIN	DATE 9-12-63
		CHECKED W P Kuchler	9-13-63
		MATERIALS	
		DESIGN W. E. Foster	9-13-63
		RELIABILITY	
P 294517	PART IDENT	W. P. Kuchler	9-13-63
P 294506	MFG INFO	PROJECT APPR.	
751501 W			

WESTINGHOUSE ELECTRIC CORPORATION — Astronuclear Laboratory, PITTSBURGH, PA.

SLEEVE, INSULATING
FUEL CLUSTER ASSY
(TITLE UNCLASSIFIED)

CODE IDENT NO.	DWG SIZE	DWG NO.
14683	B	ME-SK00158

SCALE 2/1 | WT ACT | WT CALC | SHEET 1 OF 1

4- NO MACHINING OR FINISHING PERMITTED ON O.D. & I.D. SURFACES. DIMENSION REQUIREMENTS TO BE MET BY DEPOSITION PROCESS.

3- FINISHED PART TO CONFORM TO SECTIONS 3,4,5,6 & 8 OF PDS 30014

2- BREAK ALL SHARP EDGES .015 MAX.

1- MARK ME-SK00158 & LOT SERIAL NUMBER ON CONTAINER.

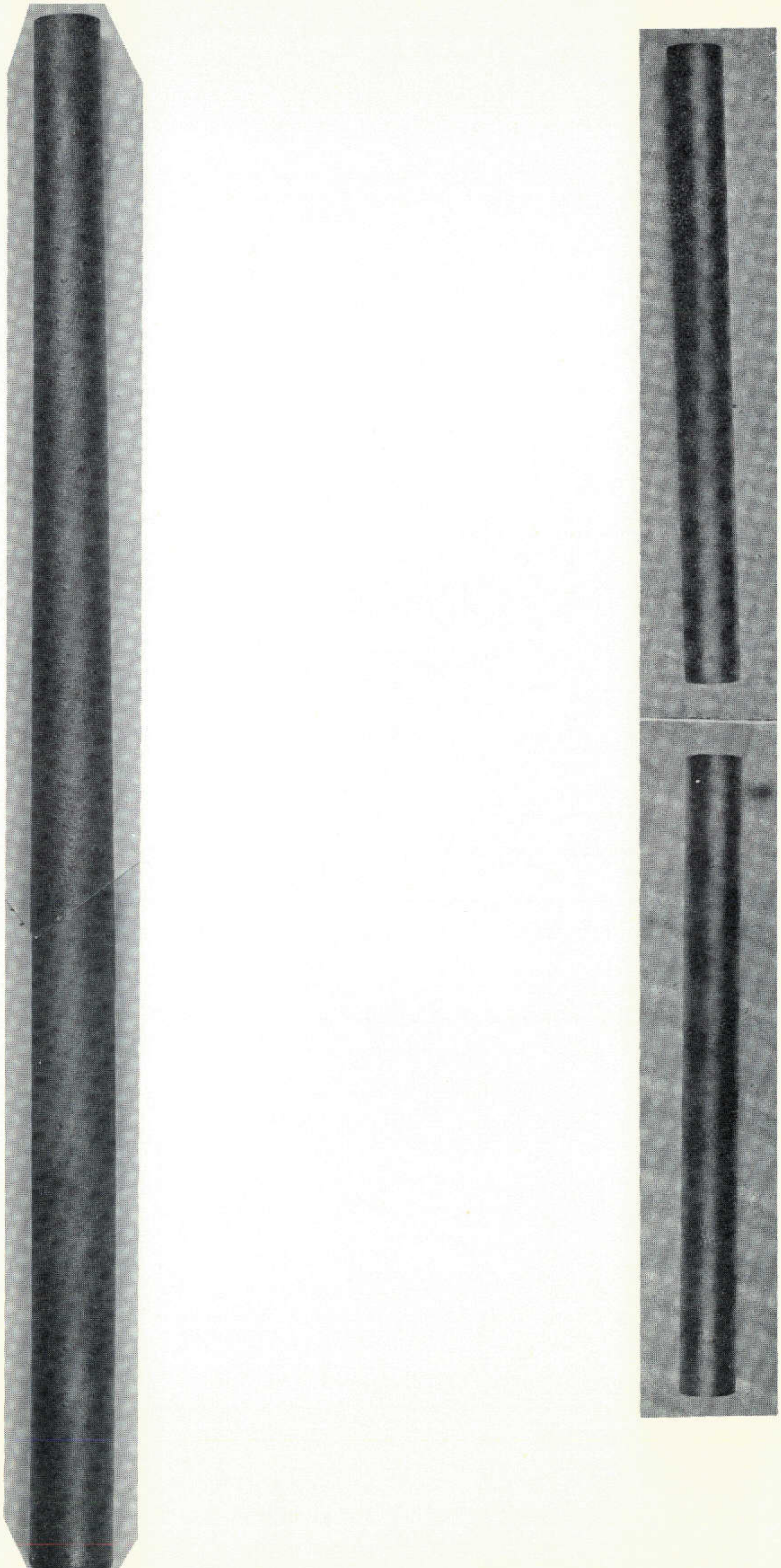


Figure 2 CONVENTIONAL AS DEPOSITED HTM BAT, WHICH POTENTIALLY YIELDS TWO MACHINED SLEEVES AS ILLUSTRATED ON RIGHT (1X)

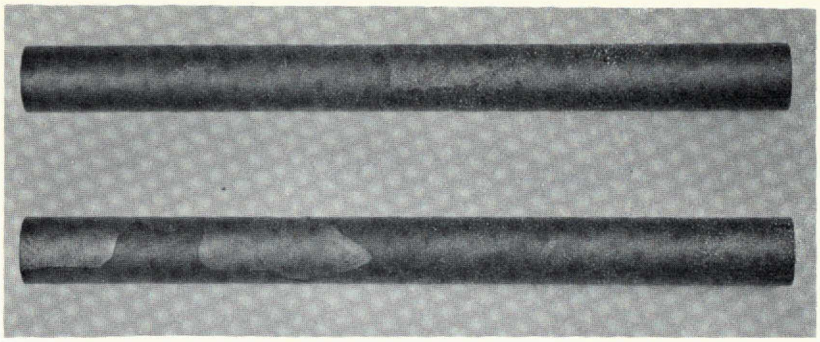


Figure 3 MACHINED SLEEVES REJECTED FOR EXCESSIVE FLAKING OR PEELING OF LAYERS AFTER BRUSHING (1X)

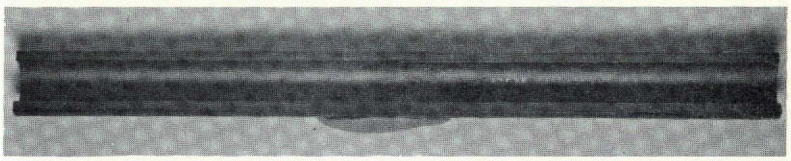


Figure 4 LONGITUDINAL CROSS SECTION OF SLEEVE IN FIGURE 3, ILLUSTRATING THE DELAMINATION SURFACE INTERSECTION AS THE CAUSE OF DESTRUCTION

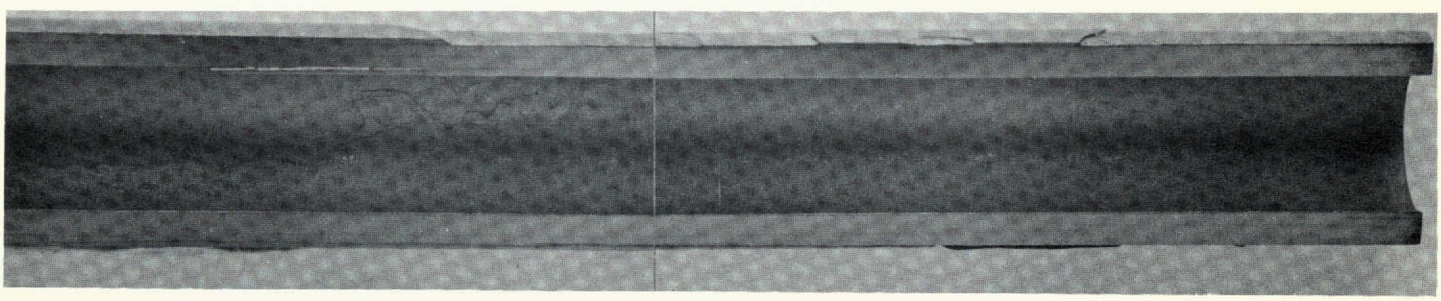
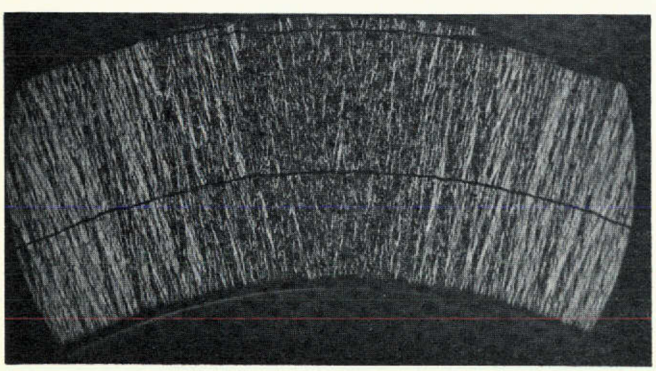


Figure 5 LONGITUDINAL CROSS SECTION OF SLEEVE IN FIGURE 3, ILLUSTRATING THE DELAMINATION SURFACE INTERSECTION AS THE CAUSE OF DESTRUCTION (3X)



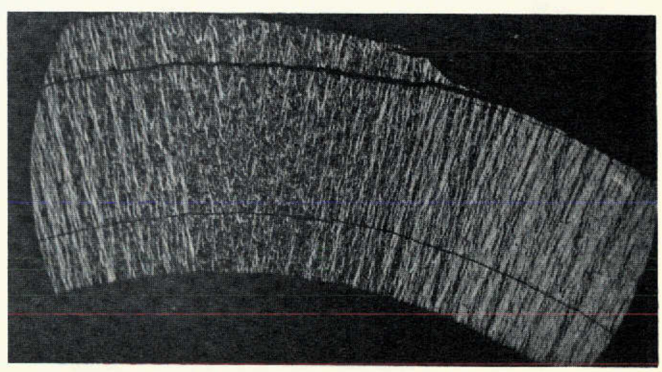


Figure 6 TRANSVERSE SECTION OF SLEEVE IN FIGURE 3 AT SPALLED AREA (25X)

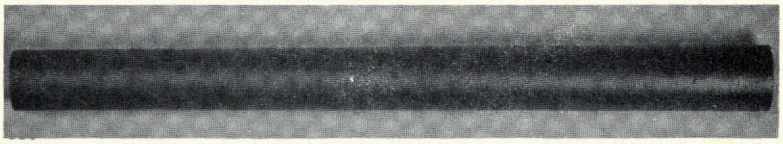


Figure 7 BRUSHED TAPERED WALL AS DEPOSITED INSULATING SLEEVE (1X)

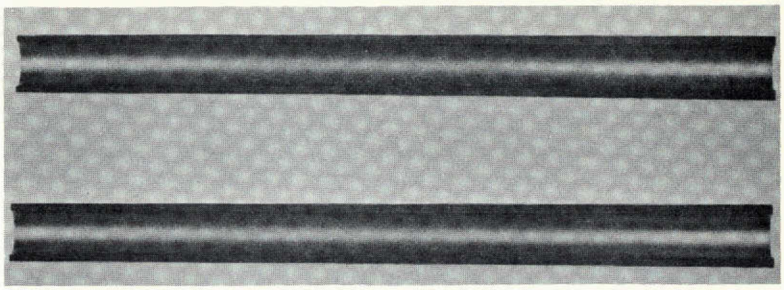


Figure 8 LONGITUDINAL CROSS SECTION OF SLEEVE IN FIGURE 7 ILLUSTRATING ABSENCE OF O.D. OR I.D. SPALLED AREAS (1X)

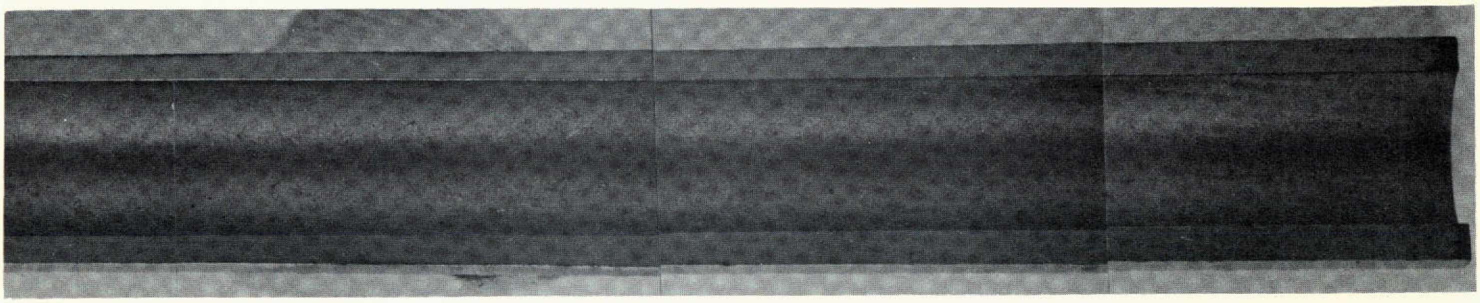


Figure 9 LONGITUDINAL CROSS SECTION OF SLEEVE IN FIGURE 7 ILLUSTRATING THE PARELLISM OF DELAMINATION TO THE ADJACENT SURFACE (3X)

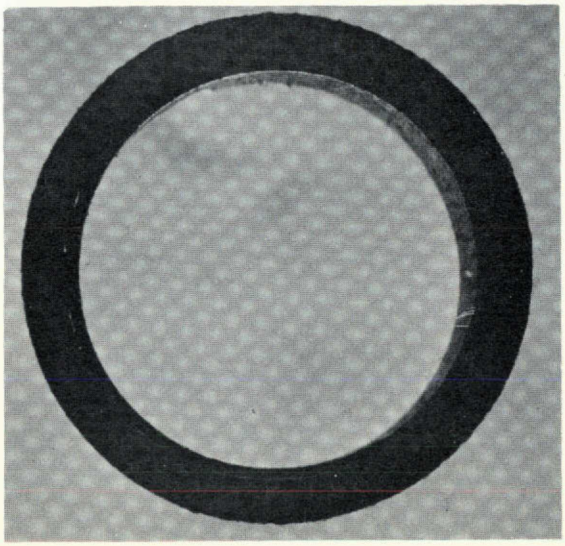


Figure 10 TRANSVERSE CROSS OF TAPERED WALL SLEEVE (9X)

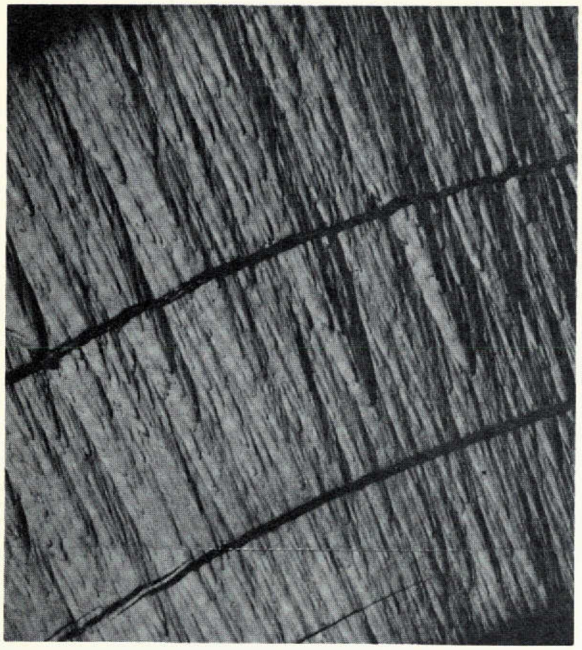


Figure 11 MICROSTRUCTURE OF TAPERED WALL SLEEVE AT LARGE DIAMETER END (50X)

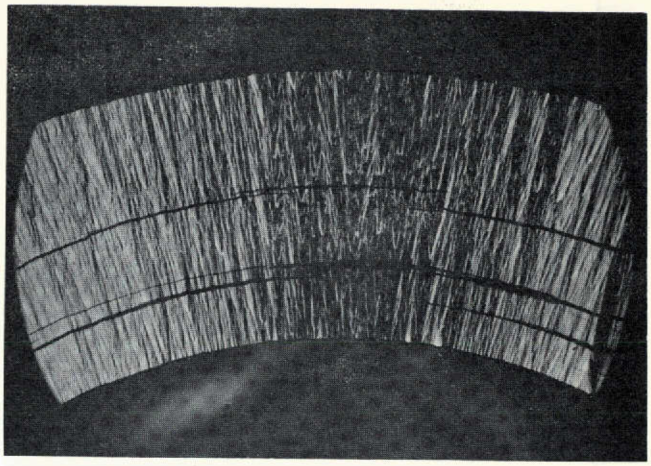


Figure 12 MICROSTRUCTURE OF TAPERED WALL SLEEVE AT LARGE DIAMETER END (25X)

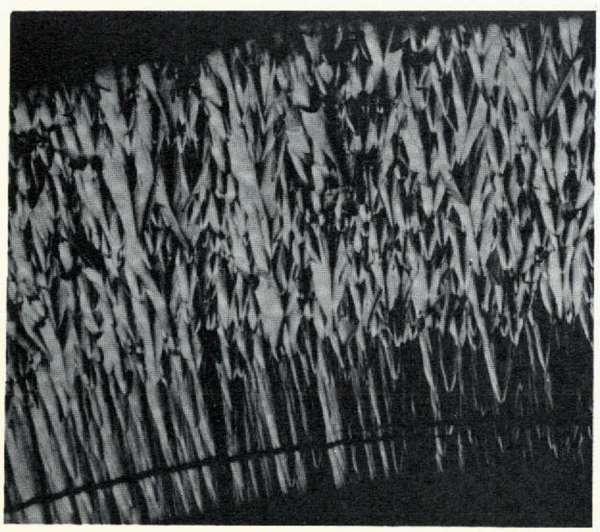


Figure 13 MICROSTRUCTURE OF TAPERED WALL SLEEVE AT SMALL DIAMETER END ILLUSTRATING CHANGE IN MICROSTRUCTURE RESULTING FROM LOCAL CHANGE IN FURNACE CONDITIONS. (50X)

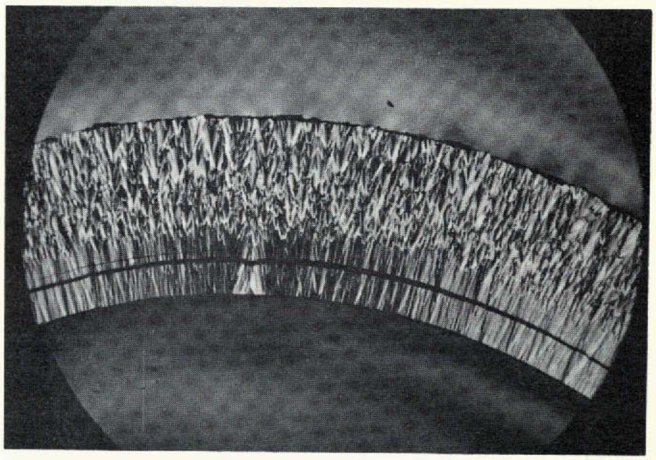


Figure 14 SAME AS FIGURE 13 ILLUSTRATING PARALLELISM OF A DELAMINATION WITH I. D. (25X)

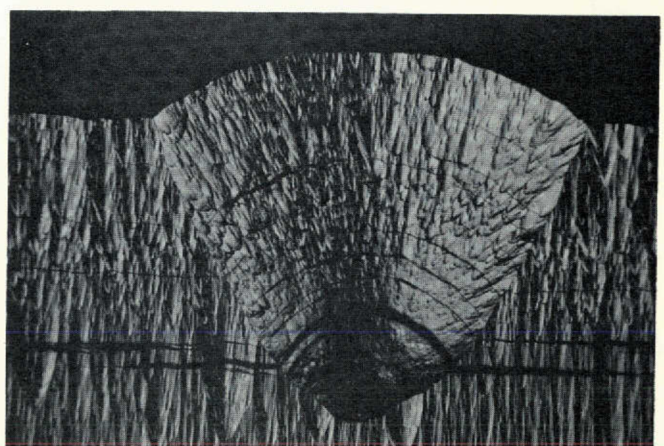


Figure 15 MICROSTRUCTURE OF EXCESSIVELY LARGE NODULE ON LARGE END ILLUSTRATING ITS INCEPTION STRUCTURAL DISTORTION, AND RESULTANT MICRO CRACKING (50X)